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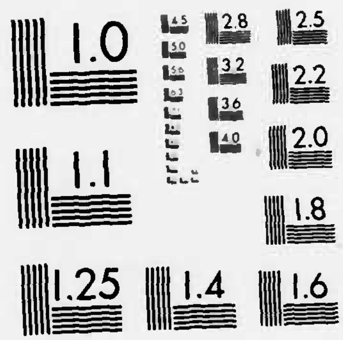
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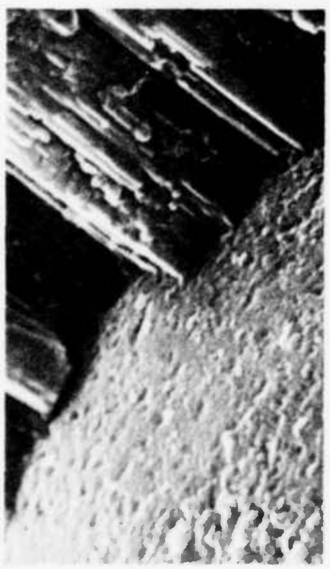

STRUCTURES LABORATORY

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MODIFICATION OF BELL CANYON TEST (BCT) 1-FF GROUT

by

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US ARMY ENGINEER WATERWAYS EXPERIMENT STATION

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Final Report

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Bell Canyon Test (BCT) 1-FF grout was developed as a candidate material for use in repository sealing applications and was actually used in two field tests in New Mexico.

This grout and modifications of it were made in the laboratory and tested or examined for workability, compressive strength, restrained expansion, permeability, phase composition, and microstructure. Most of these were done to an age of 1 year. Compressive strength and expansion data were determined beyond

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this age (960 days). Modifications included use of three other cements, two other fly ashes, a silica fume, different water contents, and different amounts of expansive additive (plaster).

Each cement and mineral admixture was characterized by conventional chemical and physical tests as well as by X-ray diffraction examination.

In general, the results indicated that the modifications to the basic BCT-1-FF grout produced other grouts that were as good as it. An exception to this was the grout mixture (M-8-C) made with shrinkage compensating expansive cement; it had an excessive flow time (>20 sec). Another grout mixture (M-9-C) also had excessive flow time and lower strength.

It was thought that these problems could be solved with more modification to these two mixtures.

PREFACE

The work described in this report was done for the U. S. Department of Energy under contract DE-AI97-81ET 46630 and modifications thereto. Messrs. Floyd Burns and Lynn Myers, Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, Ohio 43201, were earlier Project Managers. Mr. Don Moak is the present Project Manager. The basic BCT-1-FF grout mixture was developed for Sandia National Laboratories for evaluation in their Plugging and Sealing Program.

The work was done in the Structures Laboratory (SL) of the U. S. Army Engineer Waterways Experiment Station (WES) under the direction of Mrs. Katharine Mather, Project Leader at that time. Mr. A. D. Buck is now the Project Leader. Mr. Bryant Mather was Chief of the SL; Mr. J. M. Scanlon, Jr. was Chief of the Concrete Technology Division of the SL. Mr. Rhoderick and Mr. Buck prepared this report using data that were provided and analyzed by Messrs. Burkes, Reinhold, and Boa.

COL N. P. Conover, CE, was Commander and Director of WES when this work began. COL Tilford C. Creel, CE, is the present Commander and Director. Mr. F. R. Brown was Technical Director.

CONTENTS

CONVERSION FACTORS, NON-SI TO SI (METRIC) UNITS OF MEASUREMENT

Values in non-SI units of measurement may be converted to SI (metric) values as follows:

Multiply	By	To Obtain
inches	25.4	millimetres
angstroms	0.1	nanometres

MODIFICATIONS OF BELL CANYON TEST
(BCT) 1-FF GROUT

INTRODUCTION

1. The BCT-1-F grout mixture was formulated with brine as mixing water to plug boreholes in a host rock composed of salt. Since it was apparent that some host rock would be anhydrite, the BCT-1-FF grout mixture formulated with fresh water as mixing water was developed. Milestone 2 of the Structures Laboratory (SL) Geochemical Program for Fiscal Year 80[1] was the optimization of the BCT-1-FF grout mixture by the use of other cements, admixtures, modified mixture proportions, and combinations of these factors. The results of this work using four variables and evaluating these grout mixtures several ways to an age of 1 year or more are presented in this report.

MATERIALS

2. The following materials were used:

SL Serial No.	Type	Source	Date Received
	Cement		
RC-853	Class H cement	Texas	20 February 1980
RC-854	Incor Type III 0 C_3A	Texas	20 February 1980
RC-855	Type K shrinkage-compensating cement	Texas	20 February 1980
RC-857	Class H cement	Louisiana	29 May 1980
	Admixtures		
AD-536(3)	Silica fume	Alabama	16 April 1980
AD-592(4)	Fly ash	Texas	13 February 1980
AD-599	Defoamer	New Mexico	2 April 1979
AD-626	Expansive additive	New Mexico	13 February 1980
AD-627	Chemical water reducer	New Mexico	13 February 1980
AD-628	Class F fly ash	California	18 June 1980
AD-629	Class C fly ash	Wisconsin	18 June 1980

PROCEDURE

3. Chemical and physical tests were made on all the cements, the ashes, and the fume. A petrographic examination and chemical and physical tests on AD-626 and AD-627 were reported previously.[2] X-ray diffraction examination was done on all cements and pozzolans using an X-ray diffractometer with nickel-filtered copper radiation.

4. Both lime-pozzolan and cement-pozzolan cubes were made and tested for fly ashes AD-628 and AD-629 in accordance with CRD-C 256-78[3] (ASTM C 311-77). The cement-pozzolan test was made using cements RC-853 and RC-857. These data were used as a basis for selecting which cement and which fly ash to use for further testing.

5. Using the BCT-1-FF grout mixture as a basis, four variations of it were made holding the fly ash (AD-592(4)) constant. These mixtures included variations in the type of cement, amount of expansive additive (AD-626), and the water to solids (W/S) ratio. The variations were as follows:

- a. Mixture M-8-A: RC-853 as the cement, 9 percent AD-626 by weight, 0.30 W/S ratio. This was the BCT-1-FF mixture.
- b. Mixture M-8-B: RC-854 as the cement, 9 percent AD-626 by weight, 0.30 W/S ratio.
- c. Mixture M-8-C: RC-855 as the shrinkage-compensating cement, 9 percent AD-626 by weight, 0.275 W/S ratio.
- d. Mixture M-8-D: RC-853 as the cement, 7 percent AD-626 by weight, 0.275 W/S ratio.
- e. Mixture M-8-E: RC-857 as the cement, 7 percent AD-626 by weight, 0.275 W/S ratio.

6. Four other variations of the BCT-1-FF grout mixture were made while using the same cement (RC-853) and same amount of expansive additive (AD-626) with the intent that admixture or admixtures be the variable; these were:

- a. Mixture M-9-C: 30 percent solid volume replacement of the cement with the Class F fly ash (AD-628); W/S was 0.275.
- b. Mixture M-9-D: 30 percent solid volume replacement of the cement with the Class C fly ash (AD-629); W/S was 0.274.

c. Mixture M-9-E: 25 percent solid volume replacement of the cement with AD-629 and 5 percent solid volume replacement of the cement with the silica fume (AD-536(3)); W/S was 0.274.

d. Mixture M-9-F: 20 percent solid volume replacement of the cement with AD-629 and 10 percent solid volume replacement of the cement with AD-536(3); W/S was 0.274.

7. Mixture M-9-C was repeated as M-9-C-1 to reduce flow time (Table 3). More water and more water-reducing admixture (AD-627) were used; W/S was 0.280.

8. Combining materials and mixing were done by a procedure developed by the Grouting Unit of the SL. This consisted of adding the defoamer (AD-599) to the water in the bowl; dry ingredients were then added and mixing was done for 5 minutes in an N50 Hobart mixer with a wire whip instead of the usual blade type paddle. A flow cone was used to determine consistency measurements during casting of the mixtures. The flow of water was run before each sample as a reference. Flows were run according to CRD-C 79-77.[3]

9. Each mixture had the following samples cast:

a. Fifteen 2- by 2- by 2-in.* cubes for compressive strength tests at 3-, 7-, 28-, 90-, and 365-days age. At 365 days and 960 days, single cubes were broken to extend the test.

b. Two 2- by 2- by 10-in. bars for restrained expansion testing. Some were measured at 3-days age; all were measured at 7-, 28-, 56-, and at 60-day intervals; testing continued through 960 days.

c. Five 1-in.-diameter, 3-in.-long vials for X-ray diffraction and scanning electron microscope (SEM) examination at 3-, 7-, 28-, 90-, and 365-days age.

10. The cubes and the vials were sealed and subjected to accelerated curing at 38° C for 24 $\pm$ 1/2 hours. They were then demolded and stored in saturated lime water at 23 $\pm$ 1.7° C until they were used. The cubes were broken in accordance with CRD-C 227-78[3] (ASTM Designation C 109-77). The restrained expansion bars were not given any accelerated curing. Instead, they were cured and tested in accordance with CRD-C 225-76[3] (ASTM Designation C 806-75) with two exceptions. They were demolded at 24 $\pm$ 1/2 hours instead of 6 $\pm$ 1/4 hours because they were not

* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page 3.

strong enough at the earlier age. The other difference was that the bars were measured at extra ages in addition to the prescribed 7- and 28-day ages.

11. Since new permeability testing equipment was being installed when the grout mixtures were originally made, no permeability specimens were cast. These specimens were made later. These included two cylinders, each 6 in. long and 6-in. diameter, of Mixtures M-8-A, M-8-B, M-8-D, and M-8-E; the mixture with shrinkage-compensating cement (M-8-C) was not included because of its excessive flow times. Four of these specimens were tested at 7 days and four at 28-days age in accordance with CRD-C 48-73.[3] Each specimen was subjected to de-ionized water under 200-psi water pressure created by nitrogen gas acting against the water in the reservoir. In addition, two specimens each from Mixtures M-9-C, M-9-D, M-9-E, and M-9-F were cast and tested as described above except that the test ages were 8 and 28 days instead of 7 and 28 days. Mixture M-9-C with excessive flow time was chosen for testing in preference to Mixture M-9-C-1 because the latter contained very large amounts of the water-reducing admixture (AD-627) as were needed in order to reduce the flow time.

12. A vial was removed for X-ray diffraction and SEM examination of each sample at the prescribed test ages. The vials were sawed longitudinally; one-half was used for an X-ray sample and the other as a SEM sample. One-half inch from each end of the X-ray sample was sawed off leaving a 2-in.-long sample. The sawed face of the 2-in. sample was then ground in distilled water with abrasive until the saw marks were removed. The samples were examined using an X-ray diffractometer with nickel-filtered copper radiation. A vapor hood was used to surround the sample during X-ray examination in an environment of nitrogen gas and hot barium hydroxide solution to prevent carbonation and dehydration. During examination of the 28-day-old samples, the X-ray tube failed. A used X-ray tube was installed until a new one could be obtained. This tube was standardized the same way as the failed one, but due to its age, was not expected to give the same intensity. A new X-ray tube was

installed and standardized as the previous X-ray tubes. The samples examined with the used tube were held at their respective ages by freezing them in methanol. These samples can be re-examined if this becomes desirable. The SEM samples were sawed into four equal sections perpendicular to the first sawed surface. The four slices were vacuum dried at 45 to 50^{o} C for approximately 16 hours. One of the slices was selected and fractured parallel to the first sawed surface. This allowed SEM examination of a surface parallel to that of the sample that was examined by X-ray diffraction. The SEM sample was mounted on a sample stub, placed in a vacuum evaporator, and coated with about 5 nm (50 A) of carbon and about 15 nm (150 A) of a 80:20 alloy of gold and palladium.

RESULTS

13. Data for the four cements, three fly ashes, and the silica fume determined by chemical and physical tests are shown in Tables 1 and 2. Since RC-855 is a Type K shrinkage-compensating cement, it should and did differ significantly from the three portland cements (RC-853, RC-854, RC-857) in Table 1. Reference 2 provided data for the expansive additive (AD-626) and water-reducing admixture (AD-627). The defoamer (AD-599) was not tested. The higher strength of fly ash AD-629 over AD-628 by the lime-pozzolan test and by the cement-pozzolan test (Table 2) was the basis for choosing AD-629 over AD-628 for Mixtures M-9-E and F. By the same type of criteria in the cement-pozzolan test (Table 2), RC-853 was chosen over RC-857 for Mixtures M-9-C and C-1, M-9-D, M-9-E, and F.

14. Grout mixture data including bulk density, unit weights, solid volume weights, and proportions for 1.0-cu ft batches are listed in Appendix A. No two mixtures are exactly alike since any change in materials proportions always causes other changes to be made to maintain workability or some other parameter of interest. Mixture M-8-A is the same as Mixture BCT-1-FF. The water to solids ratio for the laboratory version of Mixture BCT-1-FF was 0.300. Since the two Bell Canyon field tests using this mixture at the WIPP site on 26 September 1979 and 14 February 1980[6] resulted in water to solids ratios of 0.275 and 0.272, respectively, this ratio was reduced from 0.300 to 0.275 after Mixtures M-8-A and M-8-B were cast.

15. A flow of 20 seconds or less was considered desirable for good workability; flow data for all mixtures are shown in Table 3. Mixtures M-8-C with shrinkage-compensating cement RC-855 and M-9-C with Class F ash AD-628 had excessive flow times; Mixture M-9-C was remade to reduce flow time.

16. Compressive strength data for the nine grout mixtures and one repeat mixture are shown in Table 4 for all ages. Since the differences in strengths relate to the test variables of water to cementitious materials ratio, cements, admixtures and their amounts, and amount of expansive additive, these variables need to be kept in mind as they relate to

compressive strength. For example, the much higher strength of the M-8-B mixture over the M-8-A mixture at 3 and 7 days was due to the larger surface area of cement RC-854 over RC-853 (Table 1). As expected, this difference had leveled out by 28 days. In spite of the mixture variables, strengths were generally similar with the following exceptions.

a. Mixtures M-9-C and M-9-C-1 were always low through 90 days. Since their major variable was use of the Class F fly ash (AD-628), it is obvious that one or more features of this ash were responsible for this lower strength. The higher carbon content (i.e., loss on ignition), coarseness, and higher water demand of this ash (Table 2) were probably all contributing factors to the lower strength. The slightly higher water content of Mixture M-9-C-1 would also have had some effect.

b. Mixture M-9-F failed to gain strength between 90 days and 365 days (Table 4). The reason for this is unknown since its almost companion mixture M-9-E with a little less silica fume showed normal strength development over the same period. Since M-9-F did gain strength from 365 to 960 days, the problem was not significant.

17. The results of the restrained expansion test are shown in Table 5. In general, consideration of program variables again needs to be kept in mind when examining these data. As expected, Mixture M-8-C with shrinkage-compensating cement RC-855 showed the most expansion of the 10 mixtures. Most of the mixtures showed continuous expansion except for Mixtures M-8-A and M-8-B; Mixture M-8-A showed shrinkage at the 7-day age and expansion thereafter. Mixture M-8-B showed shrinkage through 56 days and expansion thereafter. These two mixtures had more AD-626 expansive additive (9 percent) than Mixtures M-8-D and M-8-E (7 percent, which showed more expansion). The reason for this initial shrinkage is not apparent. The two bars of Mixture M-8-C made with shrinkage-compensating cement RC-855 were examined at 258-days age to determine if they showed cracking since they had expanded over 1.3 percent at the 115-day test age. Although no cracking was seen by eye, a series of narrow longitudinal cracks was detectable on bar surfaces at a magnification of 10 X. There was still increasing expansion with all mixtures at 960 days.

18. Permeability data are shown in Table 6. As expected, permeability was less with increasing age (maturity of the specimens). All of the permeability values were less than 1 x 10^{-6} microdarcies for the 16 specimens tested.

19. Examination of the unhydrated cements by X-ray diffraction showed that cements RC-853 and RC-857 contained detectable tricalcium aluminate (C_3A)* while RC-854 did not. Aside from this, the three portland cements were similar in composition. Since RC-855 was shrinkage-compensating cement, it was not directly comparable to the portland cements; it had the proper crystalline phases for this type of cement.

20. The compositions of the crystalline phases of the three fly ashes (AD-592(4), AD-628, AD-629) as determined by X-ray diffraction are shown in Table 7. In general, the compositions are usual for these materials with AD-628 containing less crystalline lime (CaO) than the other two ashes. It was apparent from handling the samples that AD-629 was finer than AD-628; this is also shown in Table 2 by the fineness data. The silica fume (AD-536(3)) was largely amorphous silica with a little crystalline quartz and possibly some crystalline silicon (Si) in it by X-ray diffraction.

21. As indicated earlier, a vial of each mixture was examined by X-ray diffraction (XRD) at different ages. Based on examination of these XRD patterns and comparisons of them within mixtures at different ages, between mixtures at common ages, and knowing that the starting crystalline or partially crystalline phases were cement, plaster, and fly ash plus or minus silica fume, the following observations were made.

Cement

22. There was always a detectable decrease in cement with time as hydration progressed. This was best followed as decreasing intensity of the 2.74+ A peak. Unhydrated cement was always present.

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* $3\ CaO \cdot Al_2O_3$.

Plaster

23. No forms of calcium sulfate (i.e., anhydrite, hemihydrate, gypsum) were usually detectable by the first examination at the 3-day age nor did they reappear. The single exception to this was that gypsum from the plaster was detectable, though decreasing, in Mixtures M-9-C and M-9-C-1 through the 28-day age. These mixtures were made with the Class F fly ash AD-628. Since these mixtures were one of the rare cases where ettringite appeared to increase with age, this suggests that the alumina from the ash that was needed to make ettringite was less readily available than from the other ashes in spite of the fact that this was the fly ash with the highest alumina content (Table 2). This assumes that all of the cements were low enough in alumina (Table 1) that a supplementary source was needed to produce enough ettringite to use all of the sulfate.

Fly Ash

24. No changes were detectable. Quartz from all three ashes was always detectable and unchanging. Mullite from ash AD-628 was also detectable in Mixtures M-9-C and M-9-C-1.

Silica Fume (AD-536(3))

25. This material was not detectable in samples examined at any of the ages.

Reaction Products

26. Ettringite was always detectable and usually seemed unchanging in amount. There were two exceptions to this; one has already been mentioned for the M-9-C mixtures; the other is that there was more ettringite in the shrinkage-compensating cement mixture (M-8-C) and it increased with age. The observation that all of the calcium sulfate was usually

gone by 3 days is taken to verify the observation that ettringite was usually constant in amount over the times involved in that there was no more sulfate left to make ettringite after 3 days.

Calcium hydroxide (CH)

27. This compound did not usually appear to change consistently in amount. Since more of it had to be made as hydration of each mixture progressed, it is believed that the combination of more CH with hydration and less CH due to combination with the ash or fume or both tended to mask changes. However, there was definitely less CH in Mixture M-9-F with 10 percent silica fume at 1 year, presumably due to combination of CH and silica fume to produce calcium silicate hydrate (CSH).

CSH

28. While this was known and observed to be present, no changes were seen due to its poor crystallinity.

Miscellaneous

29. There appeared to be a weak peak at 7.8 A in Mixture M-9-D at 1 year. This was taken to indicate that a little tetracalcium aluminate carbonate-11-hydrate (monocarboaluminate) had probably formed due to slight carbonation.

30. Aside from the few effects of materials observed, such as the effect of fly ash AD-628 on sulfate consumption and ettringite formation in Mixtures M-9-C and M-9-C-1, the effect of 10 percent silica fume on the amount of CH in Mixture M-9-F and the effect on amount of ettringite in Mixture M-8-C due to use of shrinkage-compensating expansive cement, the effects of different cements or admixtures or amounts of admixture or of water were generally not detectable by XRD. In general, the composition of the mixtures were much more similar than different.

31. Counting the repeat of Mixture M-9-C there were 10 mixtures made and tested; approximately 15 scanning electron microscope (SEM) micrographs were made of each mixture at the five ages that were examined or a total of about 150 micrographs were made at each age to study the microstructure of these mixtures. Inspection of some 750 micrographs

and comparison for selected variables indicated that in spite of the variables involved there was a general similarity of microstructure at similar ages with one exception. Mixture M-8-C with shrinkage-compensating cement RC-855 did show significantly more recognizable ettringite at early ages. As will be shown later, this microstructure becomes like that of the other cement pastes at later ages. While some variables like cement or admixture or water to cementitious solids ratio tended to modify the early age microstructure somewhat, it was the overall similarity of the microstructure that was most evident. The most striking change in microstructure was reduction in void space between 3 and 7 days to 28 days as hydration progressed. There was less striking change from 28 days on as no significant differences were readily apparent. Continued hydration leads to more densification and a uniformity of topography rather than to recognizable differences in microstructure or phase composition. Insofar as microstructure and phase composition of such mixtures are concerned, they are all evolving toward a common end point of uniformity with age rather than to differences due to variables of cement or admixture or proportions. Largely for this reason, only 4 of the total of 24 micrographs selected represent ages beyond 28 days.

32. Twenty-four micrographs representing ages from 3 days to 1 year were selected to illustrate general and special features of the microstructure of all of the grout mixtures (Figures 1 through 24). Seventeen of these show general views of microstructure at all ages and magnifications of about 200 or 2000 or 5000 or two of these, so comparisons can be made between mixtures. The other seven micrographs were included to show special rather than general features at magnifications of about 2,000 to 20,000; these seven are Figures 1, 3, 6, 8, 11, 12, and 13. The captions to the figures identify what is shown. Other comments are as follows about the cement and plaster.

a. Figures 1, 2, 4, 7, 10, and 14 show, among other things, unhydrated grains of portland cement. As indicated by Diamond,[4] Type I calcium silicate hydrate (CSH) tends to be dominant in immature cement pastes while more mature pastes typically contain Type III CSH. Both types were found in these samples with Type I being generally more abundant in the weaker (younger) samples and Type III more abundant in the stronger (older) samples.

b. Figure 3 shows typical Type I CSH, and Figure 4 shows typical Type III CSH.

c. Figure 5 shows a mixture of hydration products including some ettringite crystals in a portland cement mixture (M-8-E) while Figure 6 shows more ettringite that was typical of the shrinkage-compensating cement mixture (M-8-C). Calcium hydroxide was a hydration product also found in all mixtures.

d. Figures 5, 7, 8, 12, and 21 show calcium hydroxide. Elongated voids were found in several samples (Figures 9, 10, 21); these are believed to represent imprints of hydrous calcium sulfate crystals that pulled out during rupture or had been dissolved during hydration or both.

e. Figure 23 of the shrinkage-compensating cement Mixture M-8-C at 1 year was included to show that its microstructure is like that of ordinary cement paste by this age.

f. Figure 24 of repeat Mixture M-9-C-1 at 1 year was included to show that it has typical microstructure even though it was a less satisfactory mixture. The other reason for showing it was to suggest that the greater void space it shows may be at least partially responsible for the lower compressive strength of this mixture (Table 4).

33. The fly ash in the early age samples was visible because the grout would fracture around the fly ash spheres. In the older age samples, most of the fly ash was not visible because it was covered by hydration products, had dissolved, or had fractured through the fly ash instead of around it. Figures 7, 9, 10, 15, 16, 17, 18, 19, and 20 show fly ash spheres at early ages. Figure 12 is a good example of the contact surface between hydration products and an ash sphere. Figure 13 shows an ash sphere where it appears hydration has selectively removed glass leaving resistant crystalline phases (quartz, mullite) behind. Figure 11 shows an ash sphere that fractured during rupture of this surface.

DISCUSSION

34. The five grout mixtures of the M-8 series were generally similar even though they had differences in cement used, water to cementitious solids ratio (W/S), and amount of expansive additive (AD-626) used. Mixtures M-8-A and M-8-D both contained RC-853 cement but Mixture M-8-D had a lower W/S ratio and a smaller amount of expansive additive (AD-626). One or both of the differences caused Mixture M-8-D to be significantly stronger than Mixture M-8-A through 28 days and similar thereafter (Table 4). This strength difference was also evident in the early age microstructure as a difference in void space. Figure 15 shows more void space in Mixture M-8-A than Figure 16 for Mixture M-8-D at the 7-day age. Mixture M-8-B contained zero C_3A cement RC-854 with significantly higher surface area than the other two portland cements (Table 1). This caused Mixture M-8-B to show higher early strength at 3 days; this difference was largely or totally gone thereafter (Table 4). Mixture M-8-C with shrinkage-compensating cement RC-855 made the most ettringite of any of these mixtures by SEM at early ages and by X-ray diffraction at all ages and showed considerably more restrained expansion (Table 5). After 3 days the compressive strengths of all five mixtures became increasingly similar with time. While restrained expansions ranged considerably for Mixtures M-8-A, M-8-B, M-8-D, and M-8-E through the first 56 days, they became increasingly similar with additional age (Table 5). The permeability data were generally similar for these five mixtures (Table 6).

35. The five mixtures of the M-9 series including one repeat were also generally similar even though they contained different amounts of different admixtures. Mixtures M-9-C and M-9-C-1 had significantly lower compressive strengths at all ages tested than the other three mixtures (M-9-D, E, F) in this series (Table 4). As indicated earlier, since fly ash along with silica fume was the major variable in this series, the ash (AD-628) was obviously responsible for the lower strength of the M-9-C and C-1 mixtures (along with more water in C-1), especially when compared to Mixture M-9-D. The restrained expansion data for these five

mixtures were all positive and generally similar (Table 5). Mixtures M-9-D and M-9-E consistently showed the most expansion. As with the other series of mixtures, the permeability data were all similar and low.

CONCLUSIONS

36. Eight grout mixtures were made and compared to a ninth one (M-8-A) which was like one that had been previously tested in the laboratory and used in the field.[6] The variables were water content, cement, and amount and type of admixture. These mixtures were to be tested for flow time, compressive strength, permeability, restrained expansion, composition, and microstructure with increasing age. The results showed:

- a. Seven of the mixtures had satisfactory flow times; two were too long (M-8-C, M-9-C) in that they exceeded 20 seconds which was considered excessive.
- b. One mixture (M-9-C) and its remake (M-9-C-1) had significantly lower compressive strengths through 90 days; this was attributed largely to the Class F fly ash AD-628 that was used, partially to a slightly higher water content in the remake, and partially to lack of satisfactory consolidation of specimens. Nonetheless, all mixtures had compressive strengths in excess of 4,000 psi at 7 days, in excess of 10,000 psi at 1 year, and above 15,000 psi after 960 days.
- c. Permeability of the eight mixtures that were tested (M-8-C and M-9-C-1 were not tested) at 1- and 4-week ages were low and similar.
- d. The mixture made with shrinkage-compensating cement (RC-855) had significantly more restrained expansion than the other mixtures. Its expansion was well over 1 percent while the others were a few tenths of 1 percent at 1 year.
- e. In general, phase composition and microstructure changed with age as expected and tended to become more similar with age, also as expected.
- f. While the program variables did have recognizable effects on mixture properties at early ages, these differences tended to lessen with age.

37. In general, it appears that it is possible to modify mixture properties without any detrimental effects on composition or microstructure. Therefore, it is a matter of deciding what properties are most desirable[6] and trying to maximize them without undesirable effects on other properties of the mixture.

38. While the BCT-1-FF grout mixture (same as M-8-A) is as good as it needs to be by present criteria,[6] it can be modified to change or improve certain properties if later developments indicate this to be desirable.

39. Those mixtures having excessive flow times, M-8-C with shrinkage-compensating cement and M-9-C with fly ash AD-628, can probably be modified to have satisfactory flow times and other properties as desired.

REFERENCES

1. Paragraphs 3 and 7.2 of (FY 80) Program Plan from U. S. Army Engineer Waterways Experiment Station to Department of Energy, subject, "Investigation of Composition, Constitution, Properties, and Interactions of Materials Considered for Use in Plugging Boreholes and Shafts in Connection with Nuclear Waste Isolation."

2. USAE Waterways Experiment Station, CE, Structures Laboratory, Petrographic and Chemical Report, "Examination of Expansive Additives for Portland Cement," by A. D. Buck and T. Husbands, 11 Oct 1979.

3. USAE Waterways Experiment Station, CE, Handbook for Concrete and Cement, with quarterly supplements, Vicksburg, Miss., Aug 1949.

4. Diamond, S., "Cement Paste Microstructure - An Overview at Several Levels," Hydraulic Cement Pastes: Their Structure and Properties, Proceedings of a conference held at Tapton Hall, University of Sheffield, 8-9 Apr 1976, Cement and Concrete Association, Great Britain.

5. Barnes, B. D., Diamond, S., and Dolch, W. L., "Hollow Shell Hydration of Cement Particles in Bulk Cement Paste," Cement and Concrete Research, Vol 8, No. 3, pp 263-272, 1978, New York, N. Y.

6. Gulick, C. W., Boa, J. A., Buck, A. D., "Bell Canyon Test (BCT) Cement Development Report," SAND80-0358C, Sandia National Laboratories, May 1980, Albuquerque, N. Mex., Presented at workshop on Borehole and Shaft Plugging, Columbus, Ohio, 7-9 May 1980.

Table 1

Chemical and Physical Data for Cements* Used in Variations of BCT-1-FF Grout

Chemical Data, %	RC-853	RC-854	RC-857	RC-855
SiO_2	21.7	20.61	21.4	19.41
Al_2O_3	3.92	2.70	4.6	5.92
Fe_2O_3	4.05	5.02	4.6	3.54
MgO	3.10	2.31	0.4	0.94
SO_3	1.62	3.36	2.2	6.13
Loss on Ignition, %	0.48	0.87	0.8	0.94
Na_2O	0.14	0.06	0.46	0.27
K_2O	0.59	0.28	0.20	0.54
Alkalies - Total as Na_2O	0.53	0.24	0.60	0.62
Insoluble Residue, %	0.15	0.28	0.28	0.36
CaO	64.18	64.41	64.8	61.88
Calculated C_3S	55.62	69.49	58.0	**
Calculated C_3A	3.39	0.0	4.0	**
Calculated C_2S	20.27	6.67	18.0	**
Calculated C_4AF	12.33	0.0	14.0	**
Physical Data				
Density, mg/m^3	3.15	3.15	3.19	3.15
Fineness, Air Permeability, m^2/kg	246	415	242	496
Air Content, %	9.8	8.9	8.8	9.6
Compressive Strength, 1 day, psi	1400	2260	1640	**
Compressive Strength, 3 days, psi	2260	4180	2300	**
Compressive Strength, 28 days, psi	3030	**	3110	5840
False Set - pen. F/1, %	**	15.7	**	**
False Set Init Pen	**	30	**	**
Autoclave Expansion, %	0.05	-0.01	0.08	-0.01
Initial Set, hr/min (Gillmore)	3:25	2:05	3:10	1:05
Final Set, hr/min (Gillmore)	6:25	4:50	5:15	2:40

* Portland cements RC-853, 854, and 857 were tested in accordance with CRD-C 201-79 (ASTM C 150-78a), Reference 3. Type K expansive cement, RC-855, was tested in accordance with ASTM Designation: C 845-76T and ASTM Designation: C 807-75 (time of set). All data are averages of two or more determinations.

** Not determined.

Table 2

Chemical and Physical Data for Pozzolans* Used in Variations of BCT-1-FF Grout

Chemical Data, %	AD-592(4)	AD-536(3)	AD-628	AD-629
$SiO_2 + Al_2O_3 + Fe_2O_3$	58.5	97.7	85.8	58.6
SiO_2	32.7[(1)]	96.6[(2)]	57.87[(2)]	35.94[(2)]
Al_2O_3	16.7[(1)]	1.0[(2)]**	22.36[(1)]	16.29[(1)]
Fe_2O_3	6.4[(1)]	0.1[(3)]	5.57[(3)]	6.37[(3)]
MgO	5.6[(4)]	0.2[(2)]	1.4[(2)]	6.3[(2)]
SO_3	2.9[(2)]	0.3[(2)]	0.8[(2)]	3.4[(2)]
Moisture Content, %	0.1	0.2	0.1	0.0
Loss on Ignition, %	0.4	0.7	5.2	0.3
Na_2O	0.98[(1)]	0.45[(1)]	†	†
K_2O	0.36[(1)]	0.82[(1)]	†	†
Total as Na_2O	1.22[(1)]	0.99[(1)]	†	†
CaO	29.9[(5)]	†	4.50[(2)]	30.88[(2)]
BaO	0.69[(6)]	†	†	†
SRO	0.34[(6)]	†	†	†
TiO_2	1.58[(6)]	†	1.27[(6)]	1.05[(6)]
P_2O_5	1.04[(6)]	†	0.07[(6)]	1.02[(6)]
Mn_2O_3	0.04[(1)]	†	0.05[(1)]	0.06[(1)]
Physical Data	**AD-592(4)**	**AD-536(3)**	**AD-628**	**AD-629**
Autoclave Expansion, %	0.08	-0.06	0.00	0.06
Fineness, % Retained on 325 Mesh Sieve	16	0.42	31	15
Fineness, Air Permeability, m_2/kg	413 (e=0.500)	1917**** (e=0.714)	321 (e=0.500)	374 (e=0.500)
Lime-Pozzolan Strength, 7 days, psi	880	2050	1170	1510
Pozzolan Strength as % of Control	†	†	101††/95***	127††/124***
Water Requirement	89	†††	95	85
Density, mg/m^3	2.70	2.22	2.25	2.75

* Fly ashes AD-592(4), AD-628, and AD-629 were tested in accordance with CRD-C 256-78 (ASTM C 311-77), Reference 3. The silica fume AD-536(3) was tested in accordance with Federal Specification SSP 570 B. Supplemental tests included (a) Atomic Absorption (2) Gravimetric (3) Wet (ASTM C 595) (4) Titration (8 hydroxide) (5) EDTA (6) Plasma Emission.

** Includes Al_2O_3, TiO_2, and P_2O_5.

† Not determined.

†† Tested with cement RC-853.

*** Tested with cement RC-857.

††† Water requirement in excess of 109 percent. Flow control mix 114 percent, flow test mix 64 percent, consistency of mix soft and fluffy.

**** Extrapolation to porosity 0.500 would be much finer than that shown.

Table 3

Flow Cone Results for Variations of BCT-1-FF Grout Mixtures*

Sample No.	Flow of Water, sec	First Flow of Grout, sec**	Second Flow of Grout, sec**
M-8-A	7.6	17.8	13.6
M-8-B	7.6	14.6	15.1
M-8-C	7.6	41.6 (3 min mixing)	80.5 (5 min mixing)
M-8-D	7.5	16.7	16.2
M-8-E	7.6	15.9	15.2
M-9-C	7.6	38.1	40.0
M-9-C-1	7.4	21.6	†
M-9-D	7.5	16.1	15.8
M-9-E	7.6	17.2	16.6
M-9-F	7.4	16.7	18.9

* Done in accordance with CRD-C 79-77, Reference 3.

** Due to volume needed, the mixture was batched twice, flow was recorded, and the two batches were then combined.

† Due to extended time involved, the two batches were combined, and the flow was then determined.

Table 4

Compressive Strengths of 10 Grout Mixtures*

Sample No.	Compressive Strength, psi**					
	3 Day Old	7 Day Old	28 Day Old	90 Day Old	365 Day Old	960 Day Old
M-8-A	5790	7,330	11,490	14,750	17,400	19,100
M-8-B	9040	9,530	11,520	15,060	19,020	20,600
M-8-C	8330	9,950	11,640	13,940	18,950	20,500
M-8-D	7360	10,470	12,750	14,250	18,050	18,700
M-8-E	7190	9,000	12,720	14,810	18,000	20,500
M-9-C	4730	5,570	8,620	11,990	15,300	22,200
M-9-C-1†	3690	4,460	6,920	9,360	11,880	15,300
M-9-D	6820	8,640	11,950	14,350	16,720	17,600
M-9-E	6830	7,860	11,395	13,950	18,150	20,000
M-9-F	6700	8,360	11,750	13,260	13,100	16,800

* Tested in accordance with CRD-C 227-78 (ASTM C 109-77), Reference 3.

** Each value through 90 days is an average for three cubes; the 365-day and 960-day values are for single cubes.

† Modified repeat of Mixture M-9-C.

Table 5

Restrained Expansion Data* for 10 Grout Mixtures

Grout Mixture	Average Restrained Expansion at Ages (Days) Shown Below, %**										
	3	7	28	56	115	177	236	296	365	416	960
M-8-A	†	-0.051	0.070	0.119	0.135	0.229	0.232	0.234	0.258	0.264	0.270
M-8-B	†	-0.113	-0.052	-0.014	0.037	0.174	0.198	0.224	0.240	0.246	0.306
M-8-C	0.190	0.323	0.531	0.793	1.326	1.632	1.678	1.696	1.698	1.704	1.763
M-8-D	0.144	0.288	0.312	0.317	0.322	0.323	0.346	0.354	0.347	0.344	0.374
M-8-E***	0.099	0.135	0.210	0.215	0.220	0.222	0.233	0.237	0.235	0.235	0.240
M-9-C	0.046	0.062	0.110	0.134	0.172	0.198	0.228	0.250	0.261	0.276	0.338
M-9-C-1††	0.054	0.080	0.116	0.140	0.156	0.174	0.191	0.215	0.231	0.233	0.350
M-9-D	0.120	0.192	0.407	0.432	0.452	0.457	0.466	0.474	0.483	0.484	0.522
M-9-E	0.098	0.146	0.206	0.242	0.300	0.340	0.374	0.418	0.436	0.450	0.578
M-9-F	0.100	0.127	0.134	0.152	0.170	0.187	0.203	0.222	0.238	0.240	0.370

* Test made in accordance with CRD-C 225-76 (ASTM C 806-75) except that the bars were stripped at 24 ± 1/2 hr instead of at 6 ± 1/4 hr, Reference 3.

** Values are positive unless preceded by a minus sign. Ages after 56 days may vary a day or so.

† Not measured.

†† Slightly modified repeat of M-9-C.

*** Last four measurements are for single bar since other one became defective.

Table 6

Calculated Permeability Data for Eight Different Grout Mixtures*

Mixture Code	Permeability in Microdarcies (10^{-6})** at Ages Shown Below, Days	
	7	28
M-8-A	0.21	0.00
M-8-B	0.16	0.13
M-8-D	0.09	0.00
M-8-E	0.29	0.00
	8	28
M-9-C	0.09	0.03
M-9-D	0.00	0.00
M-9-E	0.13	0.00
M-9-F	0.06	0.00

* A 6-in.-long by 6-in.-diameter cylinder was tested for each mixture at each age using 200-psi nitrogen gas pressure on a head of de-ionized water. Mixtures M-8-C and M-9-C-1 were not tested for permeability because they were considered unusable as candidate grout mixtures for this program.

** Samples were tested according to CRD-C 48-73 with the measurements being calculated using the average flow rate for the final 5 days of the test. The samples were under pressure for 14 days.

Table 7

Phase Composition of Three Fly Ashes

by X-Ray Diffraction

Crystalline Phases	AD-592(4)	AD-628	AD-629
Quartz	X*	X	X
Mullite	X	X	n.d.
Hematite	X	X	X
Magnetite	X	X	X
Lime (CaO)	X	X	X
Anhydrite ($CaSO_4$)	X	n.d.**	X
Periclase	X	n.d.**	X
$C_4A_3\bar{S}$†	X	n.d.**	X
7.31 A material***		n.d.**	X
Iron (Fe)	?††	n.d.**	n.d.
Calcite	X	n.d.**	n.d.

* An X indicates a compound is present.

** Not detected.

† Tetracalcium trialuminate sulfate.

†† May be present.

*** May be calcium aluminoferrite which is a normal constituent of portland cement.

Figure 1. Micrograph 080280-23, X 4900. Fracture surface of Mixture M-8-D at 7-day age. Partially hydrated cement grain in center (Hadley grain, Reference 5).

Figure 2. Micrograph 072380-15, X 5300. Fracture surface of Mixture M-8-B at 7-day age. Normal hydration products and residual cement grains.

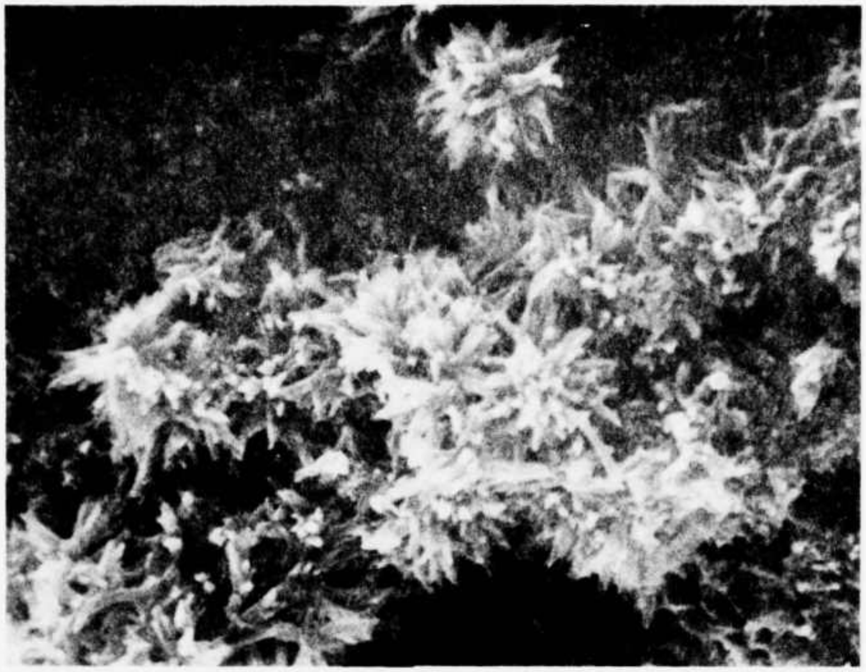

Figure 3. Micrograph 072380-20, X 10,000. Fracture surface of Mixture M-8-B at 7-day age. Clusters of Type I calcium silicate hydrate (Reference 4).

Figure 4. Micrograph 080980-40, X 2000. Fracture surface of Mixture M-9-D at 7-day age. Center is fractured residual cement grain surrounded by Type III calcium silicate hydrate (Reference 4).

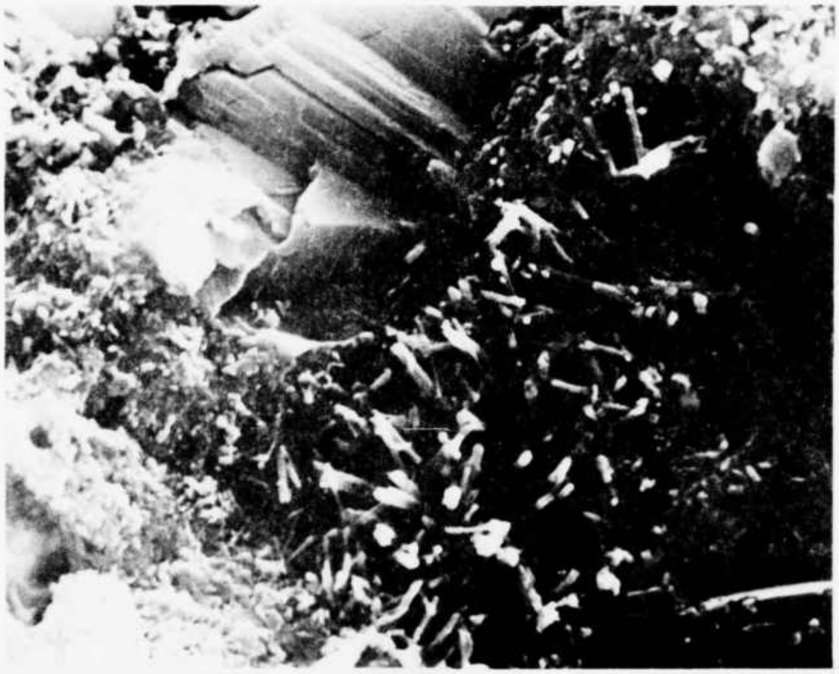

Figure 5. Micrograph 080680-26, X 5000. Fracture surface of Mixture M-8-E at 7-day age. Enlargement of part of Figure 18 shows ettringite crystals in void, calcium hydroxide (upper center), CSH, and void space.

Figure 6. Micrograph 080280-10, X 9500. Fracture surface of Mixture M-8-C at 7-day age. The abundance of ettringite in this shrinkage-compensating cement mixture is evident.

Figure 7. Micrograph 080280-33, X 1800. Fracture surface of Mixture M-8-D at 7-day age. Massive calcium hydroxide, fly ash spheres, and residual cement grains.

Figure 8. Micrograph 082680-47, X 2200. Fracture surface of Mixture M-9-C at 28-days age. Calcium hydroxide crystals in void surrounded by CSH and calcium hydroxide.

Figure 9. Micrograph 080980-3, X 2000. Fracture surface of Mixture M-9-F at 3-day age. The many elongated platy crystals are believed to be hydrous calcium sulfate from the expansive additive (AD-626). The elongated voids in this and the next micrograph (Figure 10) are believed to be imprints of these crystals.

Figure 10. Micrograph 080580-22, X 2000. Fracture surface of Mixture M-9-E at 3-day age. Residual cement grains, fly ash spheres, and hydration product. The elongated voids are believed to be imprints of hydrous calcium sulfate crystals (Figure 9).

Figure 11. Micrograph 090380-5, X 5000. Fracture surface of Mixture M-9-F at 28-days age. Broken fly ash sphere with a piece of hydration debris from the fracture surface in it. More of these spheres seem to fracture through rather than at the paste to sphere contact as hydration progresses.

Figure 12. Micrograph 083080-13, X 18,400. Fracture surface of Mixture M-9-E at 28-days age. Contact of paste hydration products and fly ash sphere (right). Massive calcium hydroxide and CSH to left; some CSH on surface of sphere.

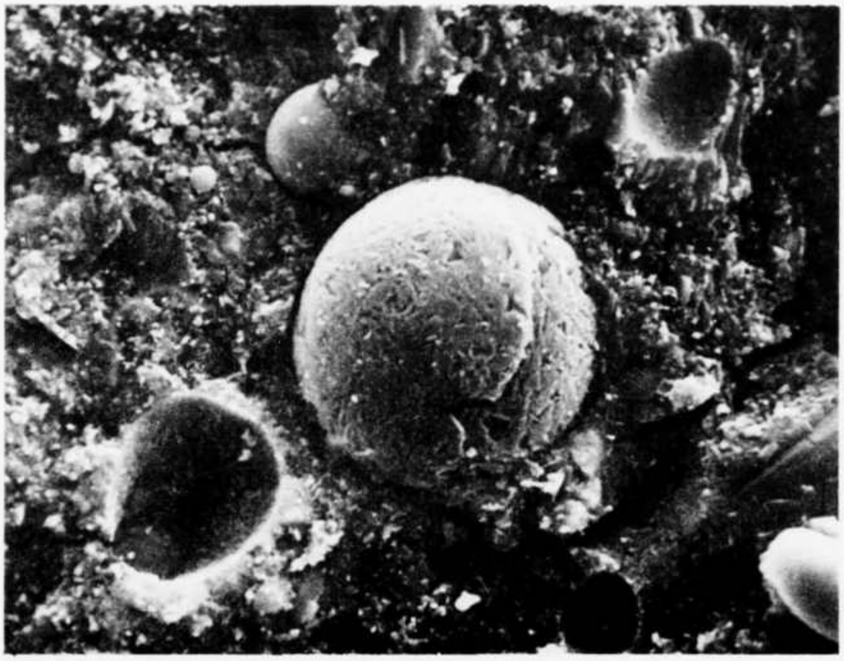

Figure 13. Micrograph 081380-6, X 2000. Fracture surface of Mixture M-8-A at 28-days age. The ash sphere in the center is believed to represent removal of glass by hydration leaving resistant crystalline phases such as quartz and mullite behind.

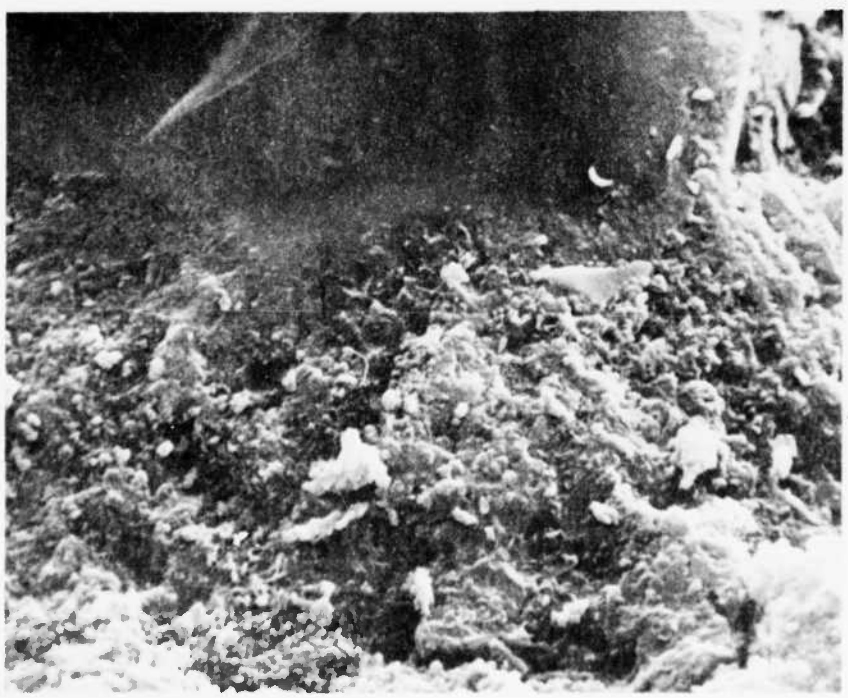

Figure 14. Micrograph 090380-12, X 5000. Fractured surface of Mixture M-9-F at 28-days age. Contact of unhydrated cement grain (top) and hydrated material (bottom). The transition across this contact is apparent. Most of the hydrated material appears to be CSH.

Figure 15. Micrograph 072380-12, X 190. Fracture surface of Mixture M-8-A at 7-day age. Surface shows more void space than Figure 16.

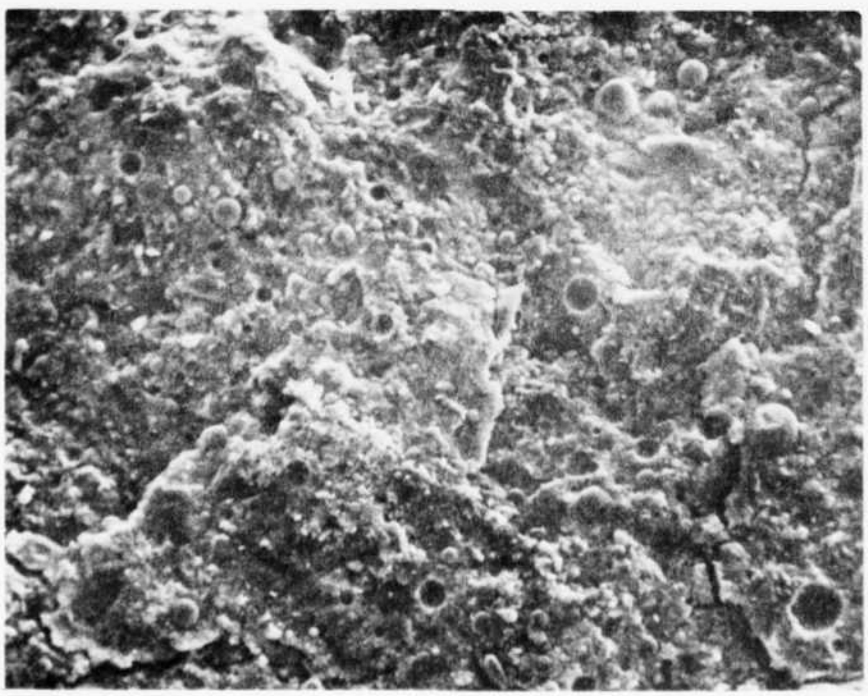

Figure 16. Micrograph 080280-32, X 180. Fracture surface of Mixture M-8-D at 7-day age. Denser surface than Figure 15.

Figure 17. Micrograph 081280-17, X 200. Fracture surface of Mixture M-9-F at 7-day age showing residual unhydrated cement, fly ash spheres, hydration product, and void space.

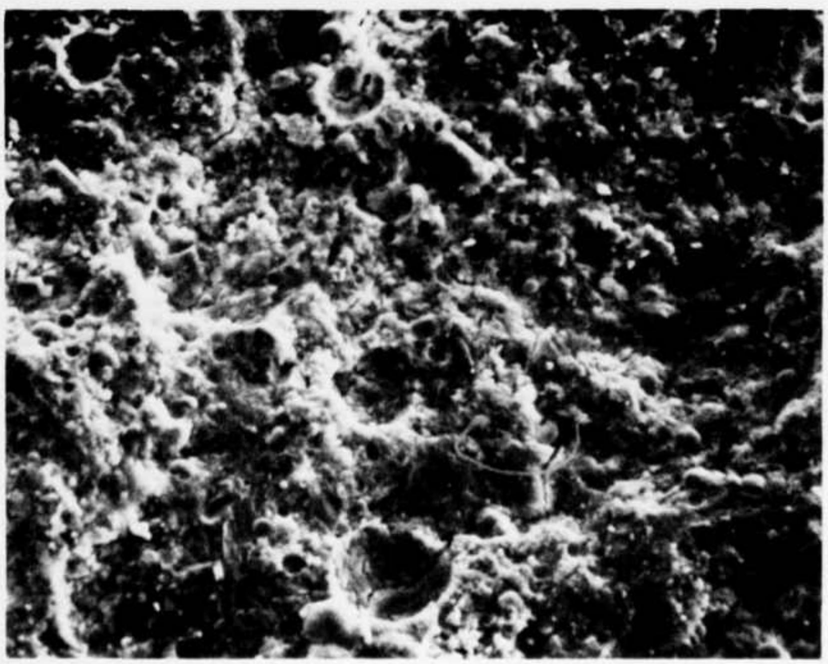

Figure 18. Micrograph 080680-34, X 200. Fracture surface of Mixture M-8-E at 7-day age showing usual appearance.

Figure 19. Micrograph 080680-18, X 200. Fracture surface of Mixture M-9-C at 7-day age. The larger size of the AD-628 fly ash spheres is shown by comparison with Figure 20 containing AD-629.

Figure 20. Micrograph 080980-48, X 200. Fracture surface of Mixture M-9-D at 7-day age. The smaller size of AD-629 fly ash spheres is shown by comparison with Figure 19 containing AD-628.

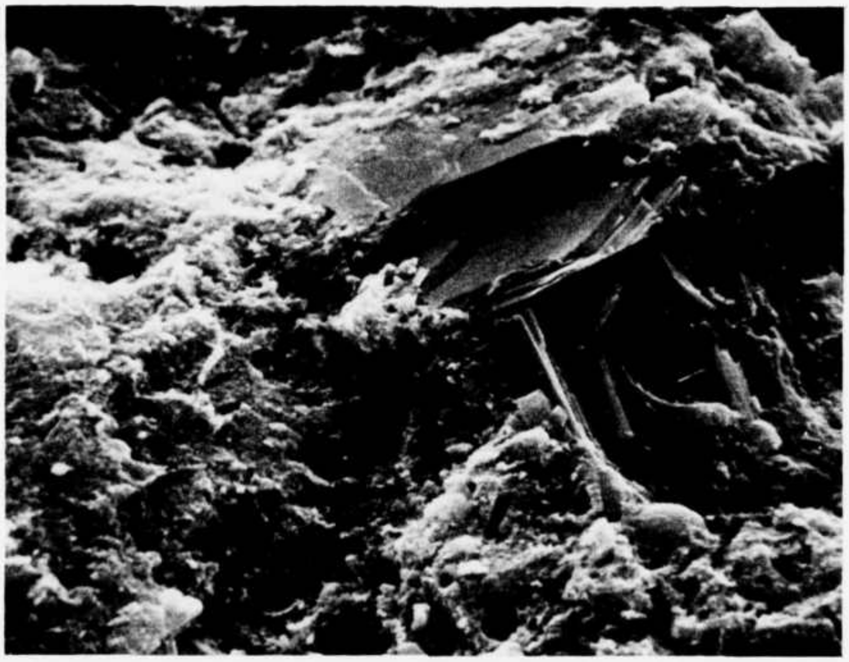

Figure 21. Micrograph 110780-10, X 2000. Fracture surface of Mixture M-9-F at 90-days age. Dense structure showing massive calcium hydroxide (center) surrounded by CSH. The number of elongated voids seen at the 3-day age (Figure 10) has decreased with time.

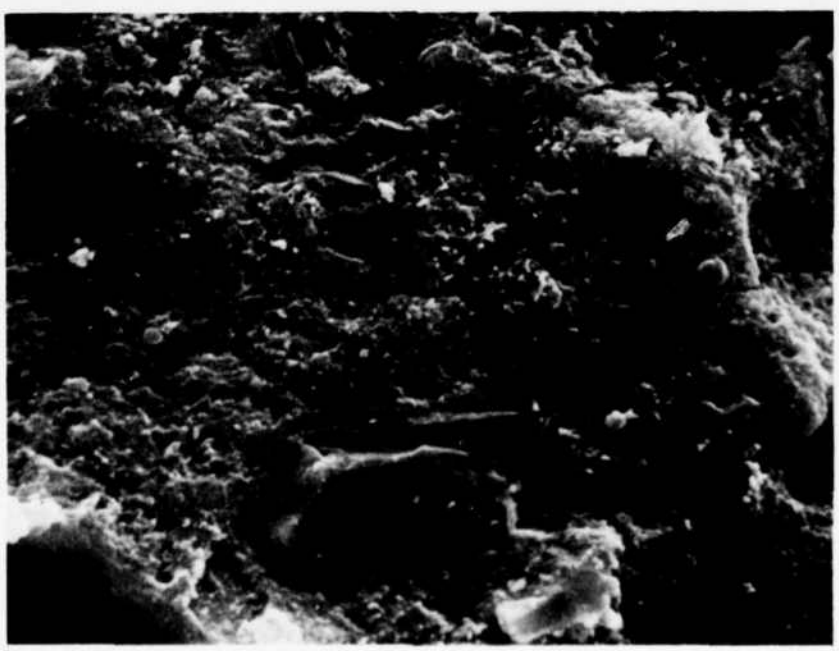

Figure 22. Micrograph 101780-37, X 1860. Fracture surface of Mixture M-8-B at 91-day age. Typical dense microstructure by this age.

Figure 23. Micrograph 072881-5, X 200. Fracture surface of Mixture M-8-C at 1-year age. Contains shrinkage-compensating cement. Most of field shows typical structure for mature cement paste. The void contains ettringite rods and calcium hydroxide plates.

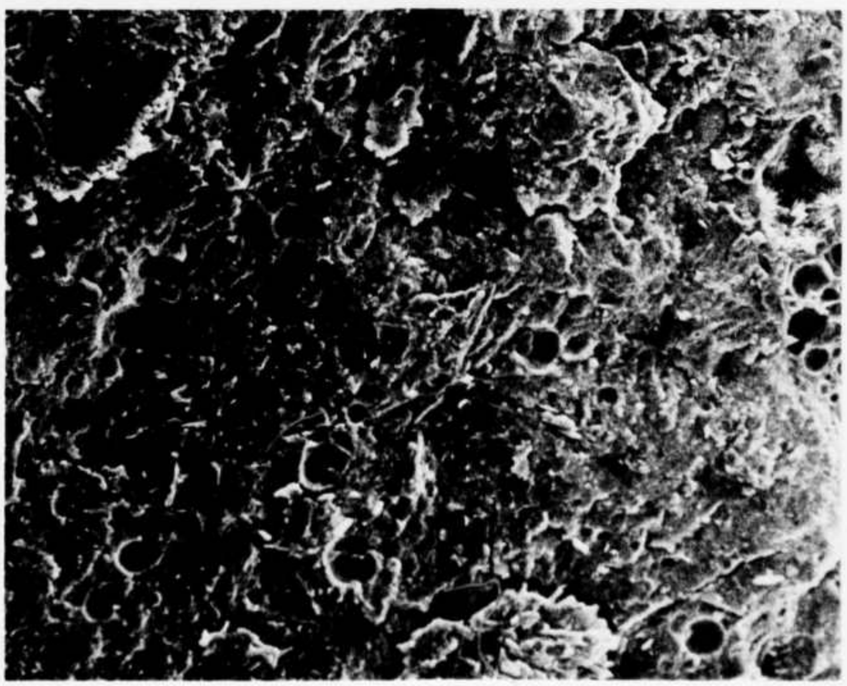

Figure 24. Micrograph 080681-22, X 200. Fracture surface of Mixture M-9-C-1 at 1-year age. Shows typical structure for mature cement paste plus a significant amount of void space due to lack of consolidation and pullout of fly ash spheres or both.

APPENDIX A: MIXTURE PROPORTIONS FOR 10 GROUTS

The following grout mixture proportions show the amounts of materials used for nine different grout mixtures and one slightly modified mixture (M-9-C-1) like M-9-C.

MIX. SER NO. M-8-A	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-592(4)	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA 1 CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0356	6.9819	9.4877	66.24	
AD-592(4)	2.70	168.21	0.0141	2.3761		22.54	
AD-626	2.70	168.21	0.0051	0.8499		8.06	
AD-627	1.58	98.43	0.0009	0.0926		0.88	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0497	3.0974		29.39	
AIR							
		AIR FREE	0.1054	13.3999		127.13	
	TOTAL	YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	
Flow 17.8			
13.6			

MIXTURE DATA

TH UW 127.13 LB/CU FT

ACT. UW ______ LB/CU FT

ACT. UW ______ LB/GAL

AIR CONTENT ______ %

BLEEDING ______ %

MIXING WATER ______ F

AMBIENT ______ F

GROUT ______ F

SETTING TIME INITIAL ______ FINAL ______

TH CF ______ B/CU YD

ACT CF ______ B/CU YD

W/C 0.44 (RC-853); 0.33 (RC-853, AD-592(4), 0.30 (RC-853, AD-592(4), AD-626); 0.30 (RC-853, AD-592(4), AD-626, AD-627)

MIX. SER NO. M-8-B	GROUT MIXTURE PROPORTIONS (WORK SHEET)	OATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-854	MINERAL AOMIX NO. 1 AD-592(4)	A. E. AOMIX
CEMENT TYPE NO. 2	MINERAL AOMIX NO. 2 AD-626	CHEMICAL AOMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL AOMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER AOMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLIO) LB/CU FT	ACT BATCH OATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.O. BATCH WT LB	FACTOR	ACT. BATCH OATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-854	3.15	196.25	0.0356	6.9819	9.3897	65.56	
AD-592(4)	2.70	168.21	0.0141	2.3761		22.31	
AD-626	2.70	168.21	0.0051	0.8499		7.98	
AD-627	1.58	98.43	0.0009	0.0926		0.87	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0508	3.1636		29.71	
AIR							
		AIR FREE	0.1065	13.4661		126.45	
	TOTAL	YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 14.6			
15.1			

MIXTURE DATA

TH UW 126.45 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEOING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME INITIAL ______ FINAL ______

TH CF ______ B/CU YO
ACT. CF ______ B/CU YO
W/C 0.45(RC-854);0.34(RC-854,AD-592(4); 0.31(RC-854,AD-592(4),AD-626); 0.307(RC-854,AD-592(4),AD-626,AD-627) WT

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

MIX. SER NO. M-8-C	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-855	MINERAL ADMIX NO. 1 AD-592(4)	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT. BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-855	3.15	196.25	0.0356	6.9819	9.8522	68.79	
AD-592(4)	2.70	168.21	0.0141	2.3761		23.41	
AD-626	2.70	168.21	0.0051	0.8499		8.37	
AD-627	1.58	98.43	0.0012	0.1146		1.13	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0455	2.8329		27.91	
AIR							
TOTAL		AIR FREE	0.1015	13.1574		129.63	
		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 41.6			
80.5			

MIXTURE DATA

TH UW 129.63 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME. INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.41(RC-855); 0.30(RC-855,AD-592(4));
0.28(RC-855),AD-592(4),AD-626);
0.274(RC-855,AD-592(4),AD-626,AD-627)

MIX. SER NO. M-8-D	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-592(4)	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0364	7.1413	9.9108	70.78	
AD-592(4)	2.70	168.21	0.0144	2.4301		24.08	
AD-626	2.70	168.21	0.0039	0.6556		6.50	
AD-627	1.58	98.43	0.0007	0.0734		0.73	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0455	2.8329		28.08	
AIR							
		AIR FREE	0.1009	13.1353		130.19	
TOTAL		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 16.7			
16.2			

MIXTURE DATA

TH UW 130.19 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME: INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.40(RC-853);0.30(RC-853,AD-592(4));
0.28(RC-853,AD-592(4),AD-626);
0.275(RC-853,AD-592(4),AD-626,AD-627)

MIX. SER NO. M-8-E	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-857	MINERAL ADMIX NO 1 AD-592(4)	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-857	3.19	198.74	0.0359	7.1413	9.9602	71.13	
AD-592(4)	2.70	168.21	0.0144	2.4301		24.20	
AD-626	2.70	168.21	0.0039	0.6556		6.53	
AD-627	1.58	98.43	0.0007	0.0734		0.73	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0455	2.8329		28.22	
AIR							
		AIR FREE	0.1004	13.1353		130.83	
TOTAL		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 15.9			
15.2			

MIXTURE DATA

TH UW 130.83 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT CF ______ B/CU YD
W/C 0.40(RC-857);0.30(RC-857,AD-592(4));
0.28(RC-857,AD-592(4),AD-626);
0.275(RC-857,AD-592(4),AD-626,AD-627)

MIX. SER NO. M-9-C	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-628	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0334	6.5507	10.5152	68.88	
AD-628	2.25	140.18	0.0140	1.9678		20.69	
AD-626	2.70	168.21	0.0051	0.8499		8.94	
AD-627	1.58	98.43	0.0009	0.0926		0.97	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0417	2.5992		27.33	
AIR							
		AIR FREE	0.0951	12.0622		126.83	
TOTAL		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 38.1			
40.0			

MIXTURE DATA

TH UW 126.83 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.40(RC-853);0.31(RC-853,AD-628); WT
0.28(RC-853,AD-628,AD-626);
0.275(RC-853,AD-628,AD-626,AD-627)

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

MIX. SER NO. M-9-C-1	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-628	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0267	5.2406	12.4378	65.18	
AD-628	2.25	140.18	0.0112	1.5742		19.57	
AD-626	2.70	168.21	0.0040	0.6799		8.46	
AD-627	1.58	98.43	0.0032	0.3185		3.96	
AD-599	0.996	62.05	0.0000	0.0016		0.02	
WATER			0.0353	2.1898		27.24	
AIR							
		AIR FREE	0.0804	10.0028		124.43	
TOTAL		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 21.6			

MIXTURE DATA

TH UW 124.43 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME: INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.42(RC-853);0.32(RC-853,AD-628);WT
0.292(RC-853,AD-628,AD-626)
0.280(RC-853,AD-628,AD-626,AD-627)

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

MIX. SER NO. M-9-D	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-629	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-626	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	AGGREGATE NO. 1	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0334	6.5506	10.2775	67.32	
AD-629	2.73	170.08	0.0140	2.3876		24.54	
AD-626	2.70	168.21	0.0051	0.8499		8.73	
AD-627	1.58	98.43	0.0012	0.1226		1.26	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0436	2.7150		27.90	
AIR							
TOTAL		AIR FREE	0.0973	12.6277		129.77	
		YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 16.1			
15.8			

MIXTURE DATA

TH UW 129.77 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.33(RC-853);0.30(RC-853,AD-629);WT
0.28(RC-853,AD-629,AD-626)
0.274(RC-853,AD-629,AD-626,AD-627)

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

MIX. SER NO. M-9-E	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-629	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-536(3)	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	Mineral Admix No. 3 AD-626	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0334	6.5507	10.3199	67.60	
AD-629	2.73	170.08	0.0117	1.9896		20.53	
AD-536(3)	2.22	138.31	0.0023	0.3236		3.34	
AD-626	2.70	168.21	0.0051	0.8499		8.77	
AD-627	1.58	98.43	0.0012	0.1226		1.27	
AD-599	0.996	62.05	0.0000	0.0020		0.02	
WATER			0.0432	2.6940		27.80	
AIR							
		AIR FREE	0.0969	12.5324		129.33	
	TOTAL	YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 17.2			
16.6			

MIXTURE DATA

TH UW 129.33 LB/CU FT
ACT. UW LB/CU FT
ACT. UW LB/GAL

AIR CONTENT %
BLEEDING %

MIXING WATER F
AMBIENT F
GROUT F
SETTING TIME INITIAL FINAL

TH CF B/CU YD
ACT. CF B/CU YD
W/C 0.41(RC-853);0.32(RC-853,AD-629) WT
0.29(RC-853,AD-629,AD-626,AD-627);
0.283(RC-853,AD-629,AD-626,AD-627);
0.274(RC-853,AD-629,AD-626,AD-627,AD-536)

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

MIX. SER NO. M-9-F	GROUT MIXTURE PROPORTIONS (WORK SHEET)	DATE
PROJECT Memorandum 2096-B		INITIALS

CEMENT TYPE NO. 1 RC-853	MINERAL ADMIX NO. 1 AD-629	A. E. ADMIX
CEMENT TYPE NO. 2	MINERAL ADMIX NO. 2 AD-536(3)	CHEMICAL ADMIX NO. 1 AD-627
CEMENT TYPE NO. 3	Mineral Admix No. 3 AD-626	CHEMICAL ADMIX NO. 2 AD-599
OTHER CEMENT	AGGREGATE NO. 2	OTHER ADMIX Water

MATERIALS AND PROPORTIONS

MATERIAL	BULK SPECIFIC GRAVITY	UNIT WT (SOLID) LB/CU FT	ACT BATCH DATA (1-BAG BATCH) SOLID VOL CU FT/BATCH	S.S.D. BATCH WT LB	FACTOR	ACT. BATCH DATA CU FT S.S.D. BATCH WT LB	ACT. BATCH WT LB OR G
RC-853	3.15	196.25	0.0267	5.2405	12.9534	67.88	
AD-629	2.73	170.08	0.0075	1.2734		16.49	
AD-536(3)	2.22	138.31	0.0037	0.5176		6.70	
AD-626	2.70	168.21	0.0040	0.6799		8.81	
AD-627	1.58	98.43	0.0010	0.0981		1.27	
AD-599	0.996	62.05	0.0000	0.0020		0.03	
WATER			0.0343	2.1391		27.71	
AIR							
		AIR FREE	0.0772	9.9506		128.89	
	TOTAL	YIELD					

MOISTURE CORRECTIONS

MATERIAL	ABSORPTION PERCENT	TOTAL MOISTURE CONTENT PERCENT	NET MOISTURE CONTENT PERCENT
Flow 16.7			
18.9			

MIXTURE DATA

TH UW 128.89 LB/CU FT
ACT. UW ______ LB/CU FT
ACT. UW ______ LB/GAL

AIR CONTENT ______ %
BLEEDING ______ %

MIXING WATER ______ F
AMBIENT ______ F
GROUT ______ F
SETTING TIME: INITIAL ______ FINAL ______

TH CF ______ B/CU YD
ACT. CF ______ B/CU YD
W/C 0.41(RC-853);0.33(RC-853,AD-629);WT
0.30(RC-853,AD-629,AD-626);
0.293(RC-853,AD-629,AD-626, AD-627);
0.274(RC-853,AD-629,AD-626,AD-267,AD-536)

WES FORM JAN 78 1589-1 EDITION OF JUN 74 MAY BE USED (OVER)

APPENDIX B: TWO ADDITIONAL MODIFICATIONS OF BCT-1-FF (M-8-A) GROUT

1. Small amounts of two additional grouts (M8A-T, M8A-T(R)) were made in October 1981. They differed from mixture M-8-A in that both contained a different Class H cement (RC-BCHSR) and mixture M8A-T(R) contained less water (0.275 W/S). The cement was a Class H obtained by blending several Class H cements from different sources; an analysis of this cement is in an unpublished WES report.* Compressive strength was determined through 28 days (Table 1). Vial specimens were examined by X-ray diffraction (XRD) and scanning electron microscopy (SEM) at 3-, 7-, 28-, and 140-days and at their 1-year ages.

2. Comparison of strength data with mixture M-8-A shows lower strength for the M8A-T and T(R) mixtures; this is probably largely due to the RC-BCHSR cement being coarser than cement RC-853. These lower strengths are similar to those for mixtures M-9-C and M-9-C-1.

3. Comparison of the XRD and SEM data between these two mixtures and with M-8-A showed the following:

- a. Three- and seven-day XRD patterns of M8A-T and M8A-T(R) showed a 7.6-A gypsum peak which was not present in the M-8-A XRD pattern at these ages. This was confirmed by the presence of crystals thought to be gypsum in SEM micrographs of M8A-T and M8A-T(R) grouts at these ages.
- b. Cavities where these crystals had been were seen in SEM micrographs at 28- and 140-days ages. These voids tended to be partially filled with laths of ettringite. These voids had been filled with cement hydration products at 1 year.
- c. There was some increase in ettringite with age. This is probably reasonable since this cement (RC-BCHSR) was coarser and would hydrate more slowly.
- d. An observation made here and oftentimes during periodic SEM examination is that while ettringite is commonly seen as elongated hexagonal crystals in young pastes or grouts, especially in voids, it is not recognizable once the samples become more massive and dense with increasing age even though XRD diffraction will show ettringite to be present.

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* A. D. Buck, J. P. Burkes, and T. S. Poole. 1983. "Thermal Stability of Certain Hydrated Phases in Systems Made Using Portland Cement."

e. XRD showed that the cement was still hydrating at 1 year.

f. The lower water content of M8A-T(R) grout was not specifically recognizable by XRD and SEM.

4. Aside from differences noted, the three mixtures were more similar than different by XRD and SEM.

5. The persistence of gypsum in these two mixtures at early ages is believed to be due to the higher sulfate content and coarseness of cement RC-BCHSR.

6. The findings for these two grouts agree with those for other modifications of BCT-1-FF grout discussed in the main body of this report.

Table B1

Compressive Strength of Two Grout Mixtures at Early Ages

Grout	Compressive Strength at Ages Shown, psi*			
	3 Days	7 Days	14 Days	28 Days
M8A-T**	3580	4760	Not Determined	6530
M8A-T(R)	4020	5360	6620	8260

* Each value is the average of three 2- by 2- by 2-in. cubes.
** W/S was 0.30.

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